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OPERATION OF THE MMC QUENCH-DEFORMATION DILATOMETER

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METALLURGICAL SECTION

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OPERATION OF THE MMC QUENCH-DEFORMATION DILATOMETER

by

L.E. Collins and J.R. Barry**

1. INTRODUCTION

The MMC Quench-Deformation Dilatometer facilitates the study of the kinetics of phase transformations in steels by accurately measuring small changes in the length of a specimen as it undergoes phase transformations. This instrument makes use of an accurately-controlled induction furnace so that transformation behaviour may be studied during heating, cooling or an isothermal hold. An hydraulically-driven deformation head may be used to deform the sample by compression and thus, allows simulation of such thermo-mechanical treatments as controlled rolling and ausforming.

A plot of dilatation versus temperature or time is produced which indicates the temperature range or time over which transformation occurs. As well, examination of the microstructure of the sample upon completion of a test provides a correlation of the microstructure with transformation behaviour. These results may be used to develop continuous cooling transformation (CCT) or isothermal transformation (IT) diagrams, or to study the effects of such parameters as alloying and deformation schedule on transformation behaviour and microstructural development.

Proper operation of the dilatometer allows a wide range of thermo-mechanical treatments to be simulated and assessed. It is the intent of this report to outline the operational procedures that have been developed at CANMET and to indicate some of the difficulties that may be encountered in attempting to simulate commercial mill practices. As well, available computer programs and their applications are summarized. The data presented are based on results obtained with a number of experimental alloys, the compositions of

which are listed in Table 1. In some cases, full analyses were not available and hence, only the principal alloying elements are listed. Although experience at CANMET is limited to steels, many of the comments should be applicable to dilatometer studies of other alloy systems.

2. THE INSTRUMENT

The measuring head of the dilatometer is shown in Fig. 1. The specimen, with a thermocouple spot-welded to it, is inserted between two ceramic platens (Fig. 1c). The right-hand platen is mounted on a spring to hold the specimen in place. Quartz rods extend from both platens either to the core or to the shell of a sensitive linear voltage differential transducer (LVDT) mounted in a frictionless suspension system. The relative displacement of the platens produced by the sample as it is heated and undergoes transformation thus generates a voltage proportional to the displacement. The LVDT has a range of ± 2.5 mm with a linear uncertainty of 0.25% full range. The output voltage is conditioned and applied to the recording devices: an X-Y recorder for ΔL - temperature measurement, a strip chart recorder for ΔL, temperature - time recording, and computer data storage. The manufacturer claims a maximum magnification of 0.1 μm/cm although in practice magnifications of approximately 2 μm/cm are being used.

Once the specimen is in place, the whole apparatus slides to the right (Fig. 1b) so that the specimen may be centered in the induction coil. The induction coil consists of two concentric coils. The outer coil is water-cooled and carries the field-inducing current, while the inner coil is used for gas cooling the specimen. A stream of quenching gas is introduced through evenly-spaced perforations on the inner face of the inner coil when high cooling rates are required. Gas flow through the inner coil is controlled by automatic or manual opening of a solenoid valve.

Compressive deformation is applied by two hydraulically driven pistons which, when triggered, drive the right hand platen towards the left hand platen which remains fixed. The pistons advance until a limit switch is tripped. A micrometer adjustment on this switch provides control over the total strain. If two deformation steps are required, a computer program is available which uses the signal generated by the LVDT to stop the head after the required initial reduction. In two stage deformation, the specimen

remains under compression between the first and second stages of deformation, the pistons being retracted only at the end of the second deformation step. At this time the pistons are retracted and the stress is removed.

During operation, the complete measuring head is enclosed in a chamber which may be either evacuated or backfilled with inert gas. The vacuum system has a diffusion pump which achieves a vacuum of 10^{-5} mbar. Operation of the dilatometer is controlled either by means of a PDP11V03 computer for which a set of standard operating programs exists, or by a temperature-function programmer microprocessor, which may be used to program non-standard thermomechanical treatments.

3. SAMPLE SELECTION AND PREPARATION

Three types of dialometer specimens are currently being used:

1. Standard
2. Rapid quench
3. Deformation

The standard specimen used for the development of CCT diagrams is 6 mm diam x 10 mm long. This comparatively large sample generates an electronic signal sufficient for very sensitive measurement of specimen length changes and usually ensures that the cross-sectional area is at least an order of magnitude larger than the grain size. This factor is important because excessive surface nucleation may markedly alter transformation kinetics.

The maximum cooling rate obtainable in the dilatometer is limited by the He gas flow rate, which determines the rate at which heat is extracted, and by the sample size, which determines the total amount of heat to be extracted. The maximum He flow rate is limited by physical dimensions of the system and thus, the sample size is the critical factor in determining maximum cooling rate. It has been found that the standard sample can be cooled at a maximum rate of ≈100°C/s (see discussion of cooling rates below). However, by drilling a 4 mm hole through the sample, leaving a wall thickness of 1 mm, the total amount of heat to be extracted is reduced; furthermore, helium may be blown on both the inner and outer surfaces. Consequently, cooling rates as high as 800°C/s may be attained with such rapid quench specimens. The major disadvantage of this type of specimen is that the 1 mm wall thickness may approximate the prior austenite grain size and thus, surface effects may play a significant role in transformation behaviour.

A smaller specimen (8 mm by 4 mm diam) is used for deformation. A smaller size is desirable to reduce the load required to deform the specimen and to ensure that a wide range of cooling rates may be achieved. Following a 50% deformation, the specimen is 4 mm in length and approximately 6 mm in diam. Because the lateral surface to volume ratios are similar, a similar range of cooling rates may be achieved with a deformation specimen deformed by 50% as with the standard specimen. The short length of the deformed sample reduces the magnitude of the signal produced by changes in length. However, this may be compensated sufficiently by the electronic amplification of the signal so that an adequate measure of the dimensional change is obtained.

All dilatometer samples must be machined so that the ends are flat and parallel. If the faces are not parallel (Fig. 2a) the specimen may not deform properly. Even if the sample is not being deformed, spurious results have been obtained because the sample moves, particularly during gas cooling. To determine if the finished faces are parallel the sample may be placed in a micrometer and held up to a light source. If a gap is evident between the sample and either anvil of the micrometer, the sample is likely to prove unsatisfactory. It is also important that the ends of the sample be flat. Small protusions (Fig. 2b) or burrs at the edges of samples (Fig. 2c) may soften at elevated temperatures and result in erroneous measurements.

The surface condition of the diameter is less critical. Because induction heating is used, large irregularities must be avoided to prevent local overheating, but no further precautions are required.

4. TEMPERATURE MEASUREMENT

Specimen temperature is measured with a Type S (Pt/Pt-10% Rh) thermocouple spot-welded to the sample. Except at very low temperatures, the effects of magnetic fields on thermocouple measurements are negligible and thus, the error introduced by the induction coil is insignificant [1]. Heat losses through the thermocouple may cause local cooling at the thermocouple junction and are of greater concern. This effect is minimized by the use of fine wires (0.127 mm) and by running the thermocouple parallel to the sample to minimize the temperature gradient. Repeated tests typically show variation of the transformation temperature within ± 5°C, indicating the good reproducibility of the results.

Problems have been encountered with temperature control at austenitizing temperatures in excess of 1200°C. The problem, characterized by transient temperature fluctuations, was the result of poor welding practice. The region of a sample in which the thermocouple wires are to be attached should be abraided to ensure a clean surface. The wires are then welded to the sample in the configuration shown in Fig. 3. The temperature will be measured at the point where the wires are closest together. Two welds on each wire will ensure that the thermocouple remains secure throughout the test. The wires should not touch if the specimen surface temperature is to be accurately measured.

To interpret the data generated by the dilatometer, it is assumed that the temperature measured by the thermocouple at the surface is uniform throughout the sample. However, in practice temperature gradients may exist in both the longitudinal and radial directions and it is worth considering whether these gradients are significant.

A longitudinal temperature gradient will arise if the platens are not perfect insulators. The thermal conductivity of the platen materials is low and thus the heat losses to the platens are expected to be small. Additionally, the insertion of thin slices of Mo foil (0.06 mm) between the sample and platen in deformation tests should further reduce heat losses, because the Mo foil will also be heated in the induction field.

Of much greater concern is the radial temperature gradient, especially at high cooling rates, when the sample is cooled by blowing He gas on the sample surface. An estimate of the temperature variation along the radius may be obtained from the following analysis. Assuming that the only temperature variation is in the radial direction, the equation for heat flow may be written in cylindrical coordinates as:

$$\frac{\partial T}{\partial t} = \alpha \left(\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} \right)$$

where T is the temperature at radius r and time t, α is the thermal diffusivity ($\alpha = k/c\rho$ where k is the thermal conductivity, c is the specific heat, ρ is the density). The sample is assumed to be initially at temperature T_o and the radiation at the surface (r = a) is governed by the boundary conditions:

$$k \frac{\partial T}{\partial r} = h (T_s - T) \quad \text{at} \quad r=a$$

where h is the coefficient of surface heat transmission and T_s is the temperature of the surroundings. The solution must also satisfy the conditions:

$$k \frac{\partial T}{\partial r} = 0 \qquad \text{at } r = 0$$

$$T = T_s \qquad \text{at } t = \infty$$

The solution to this problem has been determined by a number of authors [2,3] and may be expressed in dimensionless form as

$$\frac{T_s - T}{T_s - T_o} = 2 \sum_{n-1}^{\infty} A_n e^{-\beta_n^2 X} J_o(\beta_n \frac{r}{a})$$

where

$$A_n = \frac{k/ha}{[1+\beta_n^2 (k/ha)^2] [J_o(\beta_n)]} \qquad X = \frac{kt}{co a^2}$$

where β_n are the roots of the Bessel function which satisfy the relation $J_o(\beta) = \frac{k}{ha} \beta J_1(\beta)$; β_1, β_1, β_2, etc. being the first, second, etc. roots of the equation.

This equation has been evaluated assuming the following values for steel samples: k = 0.07 cal/cm°Cs, o = 7.3 g/cc and c = 0.16 cal/g°C. The dimensionless parameter Nu = ha/k is known as the Nusselt number and provides an indication of the rate of heat extraction. The calculation of temperature as a function of time and radius has been performed for several values of the Nusselt number which give calculated cooling rates similar to those obtained in the dilatometer.

The temperature difference between the center and the surface of a 6 mm diam sample cooled at different rates from 900°C is plotted in Fig. 4. It is seen that large temperature gradients may develop within the sample, and that the temperature difference across a sample increases rapidly with cooling rate for $t_{800-500°C}$ <30 s. At a cooling rate defined by $t_{800-500°C}$ = 9.5 s

this difference is 17°C and at $t_{800-500°C}$ = 3.8 s it becomes 40°C. Clearly, the temperature measured at the surface of a sample may be significantly different from that at the center at high cooling rates and may lead to erroneous interpretation of dilatometer results.

Several points are evident from a consideration of these calculations.

1. As a consequence of the temperature gradient, the transformation proceeds more quickly at the surface than at the center of the sample. Because the thermocouple measures the lowest temperature at any time during cooling, transformation start temperatures will be accurately determined. Transformation finish temperatures, however, are likely to be in error because the thermocouple will register a lower temperature than actually exists at the center where the transformation reaches completion last.

2. Figure 4 shows that the maximum temperature difference between the centre and the outside diameter occurs in the initial stages of cooling when temperatures are still high. For the example shown, when cooling from 900°C, the temperature difference along the radius when the surface temperature reaches 500°C is approximately one-half the maximum difference (ΔT_{max}), which occurs at temperatures greater than 800°C. Thus, the error introduced will depend upon the temperature range over which the transformation occurs. A greater error is to be expected in studying high temperature ferrite formation in a low C, low alloy steel, than low temperature martensite formation of a more highly alloyed steel.

3. In Fig. 5 the maximum temperature difference along the radius is plotted as a function of cooling rate for 6 mm and 4 mm diam specimens. At all cooling rates the temperature difference in the 4 mm diam sample is less than one-half that of the 6 mm sample. Thus, a significant reduction in the error can be achieved by simply using a smaller specimen. Similarly, a large reduction in temperature measurement errors is to be expected when hollow quench specimens are used.

At present, the effects of temperature gradients at high cooling rates have not been fully examined. The results of a more detailed study will be presented in a subsequent report.

5. COOLING RATE

With proper care, a wide spectrum of cooling rates may be obtained. The dilatometer is programmed to produce Newtonian (exponential) cooling so as to simulate the cooling behaviour which occurs when the rate is controlled solely by heat transfer at the surface, as would be the case in the air cooling large sections. It has been standard practice to express the cooling rate as the time taken to cool from 800 to 500°C. In order to obtain a full range of cooling rates, three methods of cooling have been used. For slow cooling rates ($t_{800-500°C} > 100$ s) the samples may be furnace cooled in vacuum. The use of vacuum prevents oxidation, but in very long tests, or at very high temperatures, may result in decarburization and the depletion of elements having high vapour pressure.

As the cooling rate is increased, such that $t_{800-500°C} < 100$ s, Newtonian cooling cannot be maintained to low temperatures in vacuum because the rate of heat flow from the sample in vacuum is very low, (Fig. 6). Uniform Newtonian cooling can be maintained over a limited range of cooling rates $20 < t_{800-500°C} < 100$ s by performing the test in a He atmosphere rather than under vacuum. The chamber is evacuated and then backfilled with He to a partial pressure of 16.9 kPa (25 in Hg vacuum) prior to beginning the test. This pressure is preferred to prevent arcing, yet limit the power supplied to the induction coil to achieve the required austenitizing temperature. Argon may be used instead of helium to overcome these problems, but the time to which Newtonian cooling can be maintained is longer ($t_{800-500°C} > 60$ s).

To obtain the highest cooling rates ($t_{800-500°C} < 20$ s), convectional cooling in low pressures of helium is no longer sufficient and it is necessary to direct a stream of He gas directly onto the sample surface. Dilatometer operation provides for gas cooling with or without furnace control. In our experience, the most consistent results are obtained with the furnace off. The cooling rate is adjusted by altering the gas flow rate at a constant pressure of 172 KPa (25 psi).

The cooling times obtained as a function of the gas flow rate are plotted for standard and deformation specimens in Fig. 7. It is evident that, with either Ar or He gas cooling, the reduction in cooling time decreases with increasing gas flow rate, and a minimum cooling time ($t_{800-500°C}$) of 4 s is apparently approached with He cooling. In fact, it has been found that a

minimum cooling times of 2-3 s can be achieved with He gas cooling and the gas flow rate control valve fully opened. However, the present equipment does not permit measurement of the flow rate under these conditions. In part, the cooling rate obtained is dependent upon the steel being studied (Fig. 7a) because of differences in the transformation behaviour of the steels. The low carbon steel A begins to transform above 500°C, and the heat produced during the exothermic transformation acts to slow the cooling rate and increase the total cooling time between 800 and 500°C. Steel D transforms to martensite at temperatures less than 500°C and thus the cooling time between 800 and 500°C represents the minimum cooling time that may be obtained at any gas flow rate. It is also expected that differences in the thermal properties of different steels as well as their surface condition, in particular the presence of surface oxides, may result in variations in the cooling rate obtained in any particular test. The cooling rate obtained is also very sensitive to the position of the specimen in the induction coil. Because the platens are often adjusted or replaced, it is difficult to ensure that a specimen is always located in the same position. Differences in position, likely account for differences in cooling rates among specimens of the same alloy. An example are the results obtained for deformed samples of steel A (Fig. 7b) tested at different times. For these reasons, the curves in Fig. 7 may only give an approximation of the cooling rate obtained with a particular gas flow rate. Further adjustments may be necessary depending on the alloy and the sample positioning.

6. DEFORMATION

Initially, cordierite platens were used for deformation tests because of the low thermal expansion and thermal conductivity of this material. However, the platens showed evidence of cracking after 2 or 3 tests, small pieces broke off the compressive face after only 5 or 6 tests and there was some evidence of sample adhesion to the platens. Although it was possible to regrind the face of platens, cracks formed and often extended several mm into the platens; furthermore, it proved difficult to produce parallel end faces after grinding. Pieces of Mo foil 0.06 mm in thickness, inserted between the sample and the platens failed to alleviate these problems. For this reason SiC platens, originally intended for applications requiring high compressive stres-

ses, are now used for all tests. Whereas these platens have a much longer life (≈ 150 tests), SiC has the disadvantage of having much higher thermal conductivity and thermal expansion than cordierite. Thus, significant heating has been noted in longer tests. In these cases it may not be possible to maintain Newtonian cooling at low temperatures owing to the heat stored within the platens.

Deformed samples are distinctly barrelled (Fig. 8a) because of friction between the sample and the platens; deformation is inhomogeneous and the center of the sample is more heavily deformed than the ends. Because transformation kinetics are dependent upon the total amount of strain [4,5], transformation behaviour varies with location in the sample. This effect is illustrated in Fig. 8, which compares the microstructures obtained at various positions within a sample of a microalloyed steel cooled such that $t_{800-500°C}$ = 100 s. The ΔL versus temperature plot obtained for a deformed sample is thus the total effect produced by a range of strains within a specimen, and clearly presents a problem in interpreting the effect of strain on transformation behaviour. While it must be remembered that the strain produced in rolling is also inhomogeneous, it is nonetheless desirable to eliminate friction effects from dilatometer tests.

Unfortunately, sample lubrication is a difficult problem which has yet to be satisfactorily resolved. While glasses have often been used as lubricants in hot compression testing [6,7], the use of specific glasses is limited to a narrow temperature range. The solidification of a glass as a dilatometer sample is cooled is likely to result in erroneous measurements. The selection of other lubricants is limited by the high temperatures (upwards to 1200°C) used in the dilatometer.

Strain rate can be very important, particularly in determining austenite recrystallization behaviour. Commercial rolling practice uses strain rates of approximately 10 s^{-1}. Although the strain rate applied by the deformation head of the dilatometer is variable, it has been found that if the strain rates are too high there is a loss of accuracy in controlling the total reduction. Consequently at present a strain rate of 1 s^{-1} is being used in all testing.

7. AUSTENITIZING TREATMENT

In selecting an austenitizing treatment a number of factors must be considered. Not only must the temperature be high enough to fully austenitize the steel, but the time and temperature should be selected to control the austenite grain size. Most commercial processes involve transformation of relatively fine-grained structures and thus, dilatometer simulation should avoid high temperatures and long hold times favouring significant grain growth.

With some alloys, such as microalloyed steels, it may be necessary to austenitize at temperatures sufficiently high to dissolve such precipitates as Nb(C,N). It has been the practice at CANMET to use austenitizing treatments of 5-15 min at 1150 - 1175°C for Nb-microalloyed steels. Such treatments dissolve most of the Nb(C,N), but leave sufficient precipitates to prevent large amounts of grain growth.

Austenitizing treatments which have been used for different alloys are summarized in Table 2.

8. DEVELOPMENT OF CCT DIAGRAMS

The cooling rates used to determine a CCT diagram will depend upon the nature of the diagram. In general, it is recommended that the cooling times listed in Table 3 be used to give the general shape of the diagram as illustrated in Fig. 9. Additional intermediate cooling rates can then be used to determine specific features with greater accuracy. This is illustrated in Fig. 9, where curves A-G are the recommended cooling rates and curve H ($t_{800-500°C}$ = 60 s) has been run to determine the position of the ferrite nose more closely.

Plots of sample length change (ΔL) versus temperature are analyzed in the conventional manner as discussed by Eldis et al. [8]. The start and finish of transformation are taken to be the temperatures at which deviation occurs from approximately linear thermal expansion of the austenite and transformation products as illustrated in Fig. 10. Often more than one transformation product is evident. In the simplest case the transformations are well separated and easily distinguished, Fig. 10. However, in many cases, a change in transformation behaviour is marked only by a small change in the slope of the ΔL versus T curve, as is the case in Fig. 10 in which the ferrite transformation gives way to pearlite formation at lower temperature.

The formation of pearlite is particularly difficult to distinguish from ferrite in low carbon steels where the pearlite content is low. In practice, the region of pearlite formation is only accurately determined by quenching out samples from various temperatures on the cooling curves and examining the microstructure.

The results obtained at various cooling rates are accumulated in a CCT diagram in which the actual temperature-time measurements are plotted on a log time scale. The point at which the temperature passes through Ac_3 is normally taken as the zero time. The transformation start and finish temperatures, as determined from the plots of ΔL versus T at each cooling rate are plotted and connected by smooth lines to indicate the regions of formation of different transformation products. A complete CCT diagram should also indicate the Ac_1 and Ac_3 temperatures as discussed below, as well as the austenite grain size prior to transformation and the deformation history. It is often of value to provide an indication of the progress of the transformation by including isotransformation contours indicating the temperatures at which 10%, 25%, etc. of the austenite has transformed. The determination of the fraction transformed from the ΔL versus T curves is discussed below.

9. DETERMINATION OF Ac_1 AND Ac_3

The temperature at which austenite begins to form during heating is referred to as Ac_1 and the temperature at which the ferrite to austenite transformation is completed during heating is termed Ac_3. These represent the maximum temperatures at which transformation could begin and reach completion upon cooling and are always indicated on CCT diagrams. Because the measured temperatures are dependent upon the heating rate, it has been standard procedure to determine these temperatures at a constant slow heating rate of 2 to 3°C/min [8]. It is the practice at CANMET to heat the sample rapidly to 500°C and to then heat at 2°C/min to 1000°C. This method provides a sufficient measure of thermal expansion of low temperature phases and austenite so that critical temperatures can be determined readily, while limiting the total time taken for a test. This heating cycle is accomplished by the dilatometer microprocessor with the program listed in Appendix I. Upon reaching 1000°C this program immediately quenches the sample to room temperature.

10. CALCULATION OF THE FRACTION TRANSFORMED

While determination of transformation start and finish temperatures is valuable in itself, these temperatures may be significantly higher and lower respectively, than the temperature range over which the bulk of the transformation occurs. In this case, it is of value to know the fraction of transformation which has been completed at any given temperature. This information may be determined directly from the ΔL versus T plot. As described by Eldis et al. [8], the fraction transformed, X, is determined by extrapolating the curves for the thermal contraction of the austenite and of the transformation products, as shown in Fig. 11, and calculating the ratio of the lengths AB and AC.

$$X = AB/AC$$

This calculation involves a number of assumptions:

1. The thermal expansion of the austenite varies linearly with temperature.
2. The thermal expansion of all transformation products is the same and varies linearly with temperature.
3. The expansion accompanying the austenite transformation is the same regardless of the transformation product.

While some error is introduced through these assumptions, the method provides sufficient accuracy for most applications.

11. AVAILABLE PROGRAMS

In the following, the computer programs available for dilatometer operation and their application are briefly discussed. Other thermomechanical treatments may be achieved by the microprocessor for which the necessary programs are developed as required.

DIL

This program is used for determining transformation behaviour in cases where no deformation is required. The sample is austenitized at the desired temperature (T_1), and time and cooled under controlled conditions, Fig. 12.

DEF

The DEF program is used to study deformation effects on transformation behaviour. A sample is austenitized (T_1) and may then be deformed at two different temperatures (T_2 and T_3) by reductions totalling 50% or less of the initial length. An isothermal hold at the deformation temperatures T_2 and T_3 may follow each deformation. After the hold at T_2 the sample is quickly cooled (≈75°C/s) to the second deformation temperature.

In selecting the length of the isothermal holds following deformation, consideration should be given to the effect of the treatment on recrystallization and precipitation and to the schedule that one is attempting to simulate. It has been our practice in work with low alloy steels to allow 15 s after the initial deformation to simulate the interpass time in typical rolling schedules. In most steels, this is sufficient time for complete static recrystallization, unless recrystallization is inhibited by precipitation of microalloying elements. In most rolling schedules, cooling begins immediately after the final pass and consequently, the hold time at the second deformation temperature in the dilatometer simulation is kept short. It has been found that a 3 s hold allows sufficient time for the temperature to stabilize and deformation to be completed prior to cooling.

DEF2

This program is similar to DEF except that the isothermal holds following deformation (at T_2 and T_3) are eliminated and instead, the cooling rates from the austenitizing temperature, T_1 to T_2 and from T_2 to T_3, are controlled. As well, the controlled cooling following completion of deformation may be divided into two segments (T_3 to T_4 and T_4 to T_5) with different cooling rates.

Controlled cooling within the austenite region offered by this program has the advantage that it more closely simulates actual rolling practice, but, from an experimental point of view, recrystallization and precipitation processes no longer occur isothermally and thus, are more difficult to characterize.

ISO

Isothermal transformation (IT) diagrams may be generated with the ISO program. Samples are austenitized at temperature T_1, rapidly quenched (≈ 5 s)

to the desired temperature T_2, and held the required length of time. A sample may be quenched to room temperature, or cooled at a controlled rate following an isothermal hold.

WELD

This program is used to simulate the thermal cycle experienced in a weld heat affected zone (HAZ). The sample may be rapidly heated (heating times as short as 6 s have been attained) to the peak temperature, T_1 in Fig. 12, and then cooled immediately at a controlled rate.

To achieve the maximum heating rate, hollow rapid quench samples must be used and tests must be performed in vacuum or Ar atmosphere. Heat losses in a He atmosphere are such that high heating rates cannot be maintained to high temperatures ($\geq$1000°C). It has also been found that it is necessary to vary the parameters input to the program depending on the cooling rate to be achieved and the method necessary to achieve it. Table 4 summarizes the parameters used in an HAZ simulation in a microalloyed steel heated to 1350°C in 6 s and cooled at various rates. The peak temperature in these tests varied by ±10°C.

REFERENCES

1. Manual on the use of Thermocouples in Temperature Measurement, ASTM, STP 470, (1973).

2. H.S. Carslaw and J.C. Jaeger, Conduction of Heat in Solids, 2d ed., Oxford University Press, London (1959) p. 202.

3. A.B. Newman, Trans. Amer. Inst. Chem. Engrs., 27 (1931) 203.

4. D.J. Walker and R.W.K. Honeycombe, Metal Sci., 12 (1978) 445.

5. L.E. Collins, J.R. Barry and J.D. Boyd, Symposium on Phase Transformations in Ferrous Alloys, Ed. A.R. Marder and J.I. Goldstein, AIME, (1984) 397.

6. M.G. Akben, I. Weiss and J.J. Jonas, Acta metall., 19 (1981) 111.

7. K. Titto, G. Fitzsimons and A.J. DeArdo, Acta metall., 31 (1983) 1159.

8. G.T. Eldis, Hardenability Concepts with Applications to Steel, Ed. D.V. Doane and J.S. Kirkaldy, AIME, (1978) 126.

Table 1 - Steel compositions (wt %)

Steel	C	Mn	Mo	Ni	Cr	Nb	V	Ti	S	Si	Cu	N	Al
A	0.11	1.46	-	-	-	.055	-	-	.002	.28	-	.0069	.02
B	0.07	1.42	.24	.013	.21	.053	.05	.012	.004	.28	.02	.008	.024
C	0.47	0.93	.018	.11	.11	-	-	-	.003	.23	.28	?	.031
D	0.27	0.63	.3	-	3.39	-	.03	-	?	?	.13	?	?
E	0.45	0.70	1.00	.50	1.00	-	.10	-	?	?	-	?	?
F	0.9	?	.23	.19	1.00	-	-	-	?	?	?	?	?
G	0.33	1.44	.018	.11	.11	-	-	-	.003	.23	.28	?	.031

Table 2 - Austenitizing treatments

Alloy	Starting condition	Aust. Time (min)	Aust. Temp (°C)	Final γ grain size (μm)	Purpose
A	As-rolled plate	5	1150	35.7	-Dissolve Nb(C,N) precipitates while maintaining a fine austenite grain size
	As-rolled plate	15	1150		
	As-rolled plate	5	1175		
	As-rolled plate	15	1175		
B	As-rolled plate	15	1200	39	-Same as Steel A. -Ti-bearing steel can be exposed to higher temperature to ensure Nb(C,N) dissolution without excessive grain growth.
C	As-rolled plate	5	960	≈15	-Obtain fine austenite grain size as would be expected at the finish of rolling.
D	As-rolled plate	20	870		-Ensure dissolution of Cr carbides.
E	As-rolled plate	20	870		-Ensure dissolution of Cr carbides
F	As-rolled plate	10	927	≈20	

Table 3 - Standard cooling rates for development of CCT diagrams

$t_{800-500°C}$ (s)	Curve (Fig. 9)
3	A
10	B
30	C
100	D
300	E
1000	F
3000	G

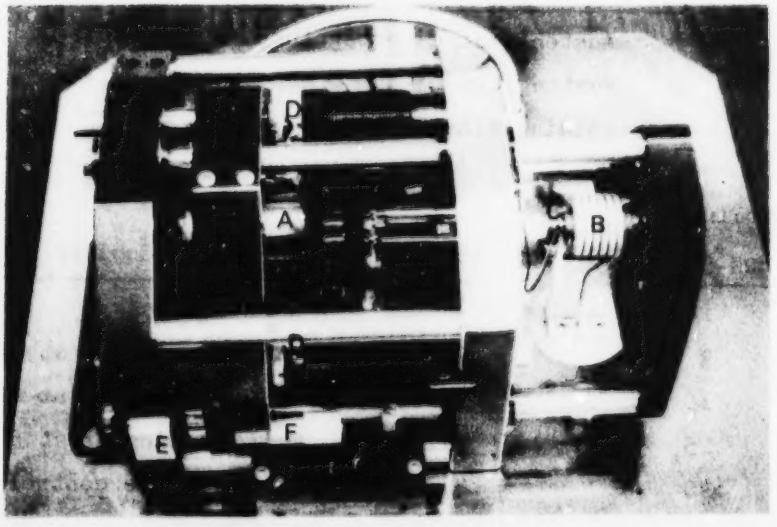
Table 4 - Computer input for HAZ simulation

Austenitizing temperature = 1350°C
Heating time = 4 s
Holding time = 9 s

Cooling time $t_{800-500°C}$ (s)	Atmosphere	Cooling method
3	Vacuum	flowing Ar at maximum rate
10	Vacuum	flowing Ar at 20 scfh
25	Vacuum	flowing Ar at 5 scfh
>40	Vacuum back-filled to 25 in Hg with Ar	Furnace control

Note: These tests were performed on irregularly-sized samples 10 mm long x 5 mm diameter with a 3 mm diam hole.

Fig. 1(a). MMC Quench deformation dilatometer. (neg 9202)



(b). Measuring head. (neg 9202)
Fig. 1. cont'd.

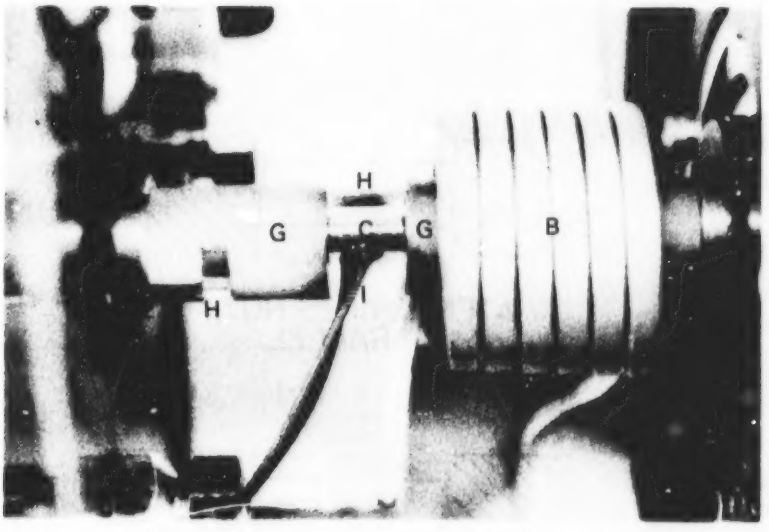


Fig. 1(c). Closeup of Fig. 1(b) showing specinen positioning. (neg 9202)

Components of measuring head

A. LVDT
B. Induction coil
C. Specimen
D. Deformation pistons
E. Limit switch
F. Micrometer adjustment of limit switch
G. Platens
H. Extension rod to LVDT
I. Thermocouple.

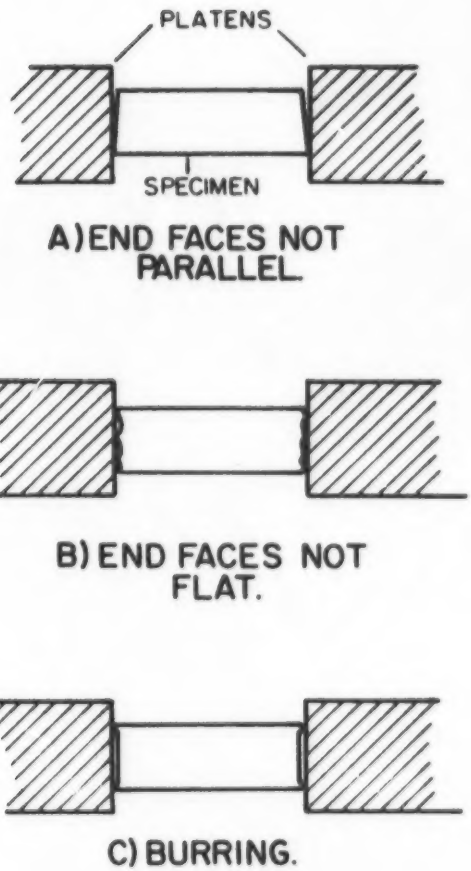


Fig. 2. Illustration of improperly prepared specimens. (neg 10669)

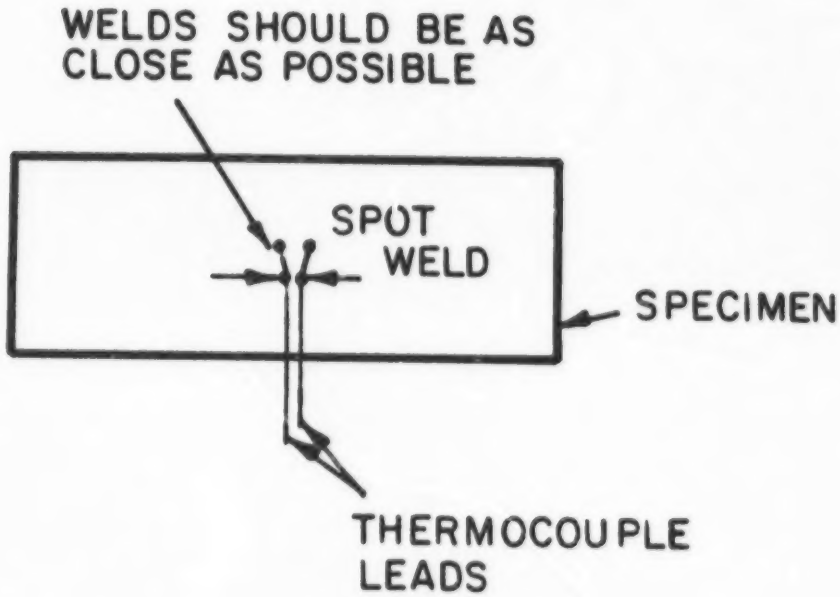


Fig. 3. Configuration of thermocouple spot-welded to the specimen. (neg 10672)

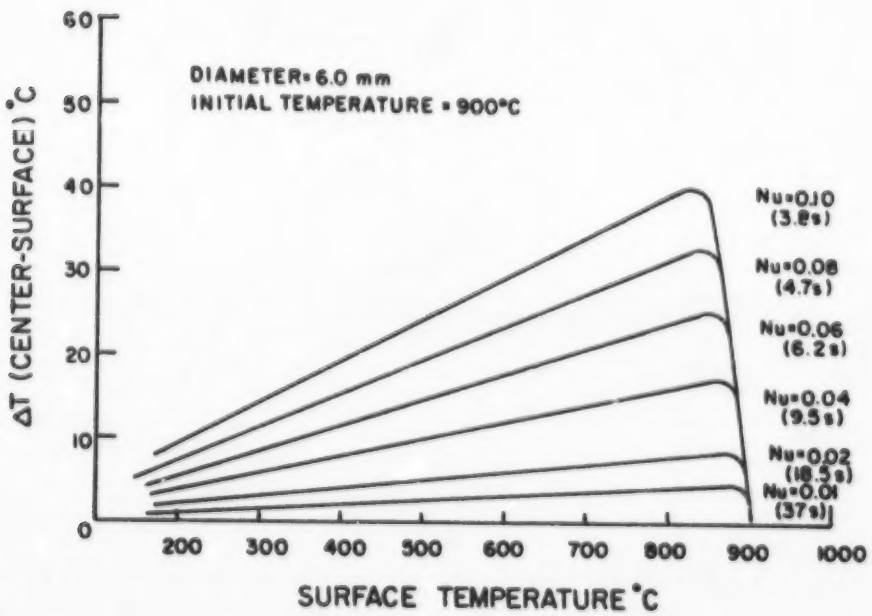


Fig. 4. Plot of calculated temperature difference between center and surface of a specimen. (neg 10664)

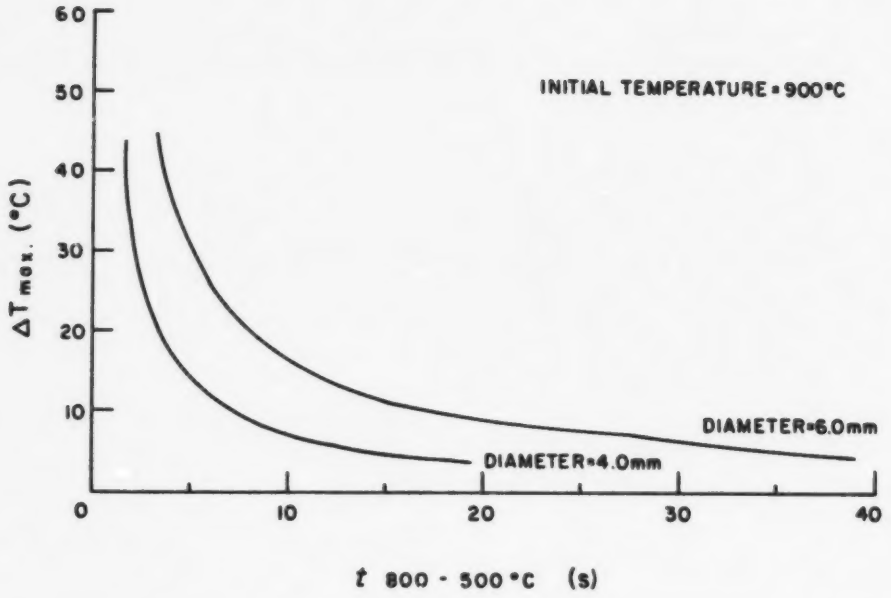


Fig. 5. Plot of maximum temperature difference versus the surface cooling rate. (neg 10663)

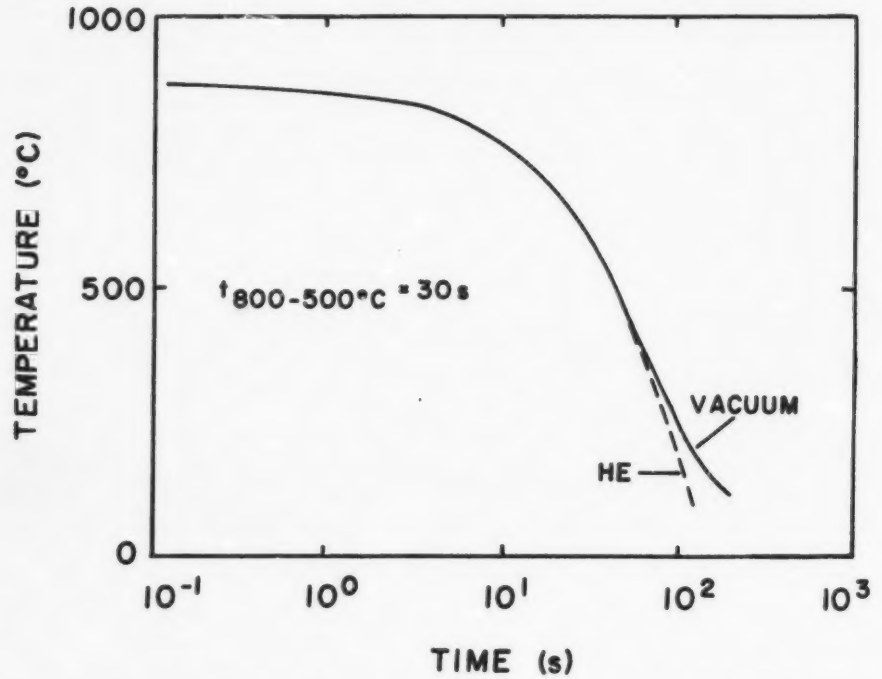
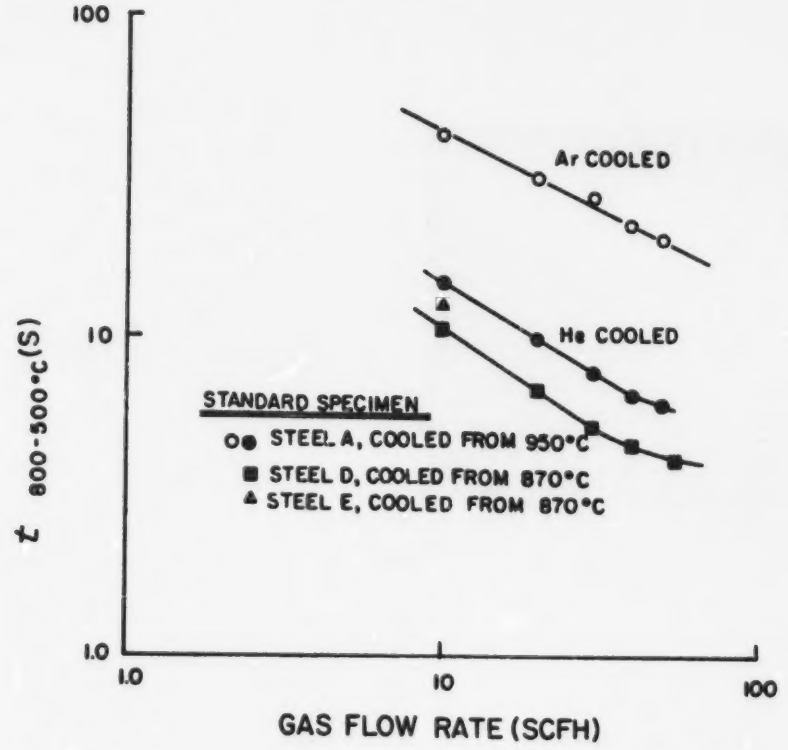


Fig. 6. Comparison of cooling in vacuum and partial He atmosphere (25 mm Hg). (neg 10670)



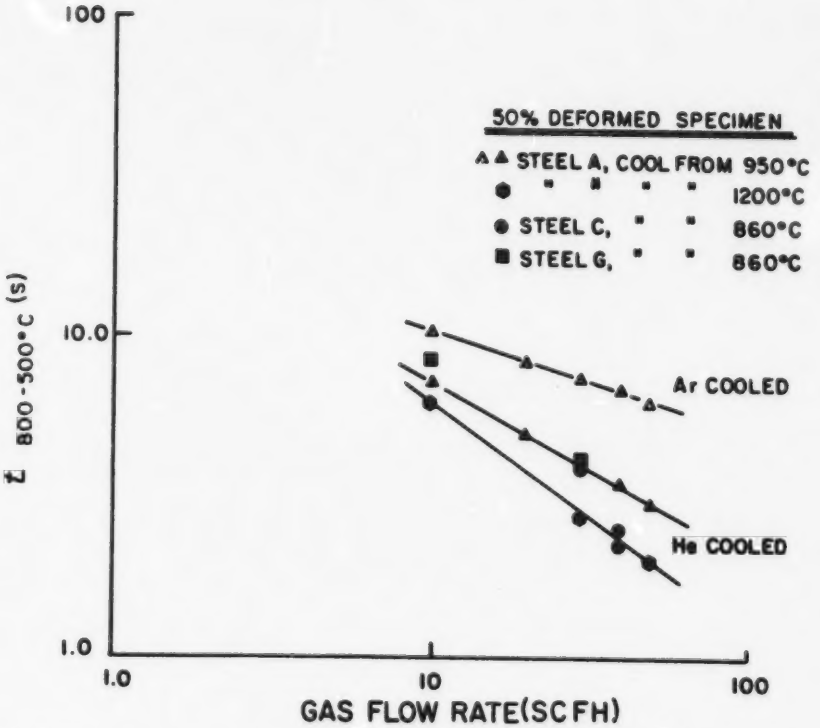
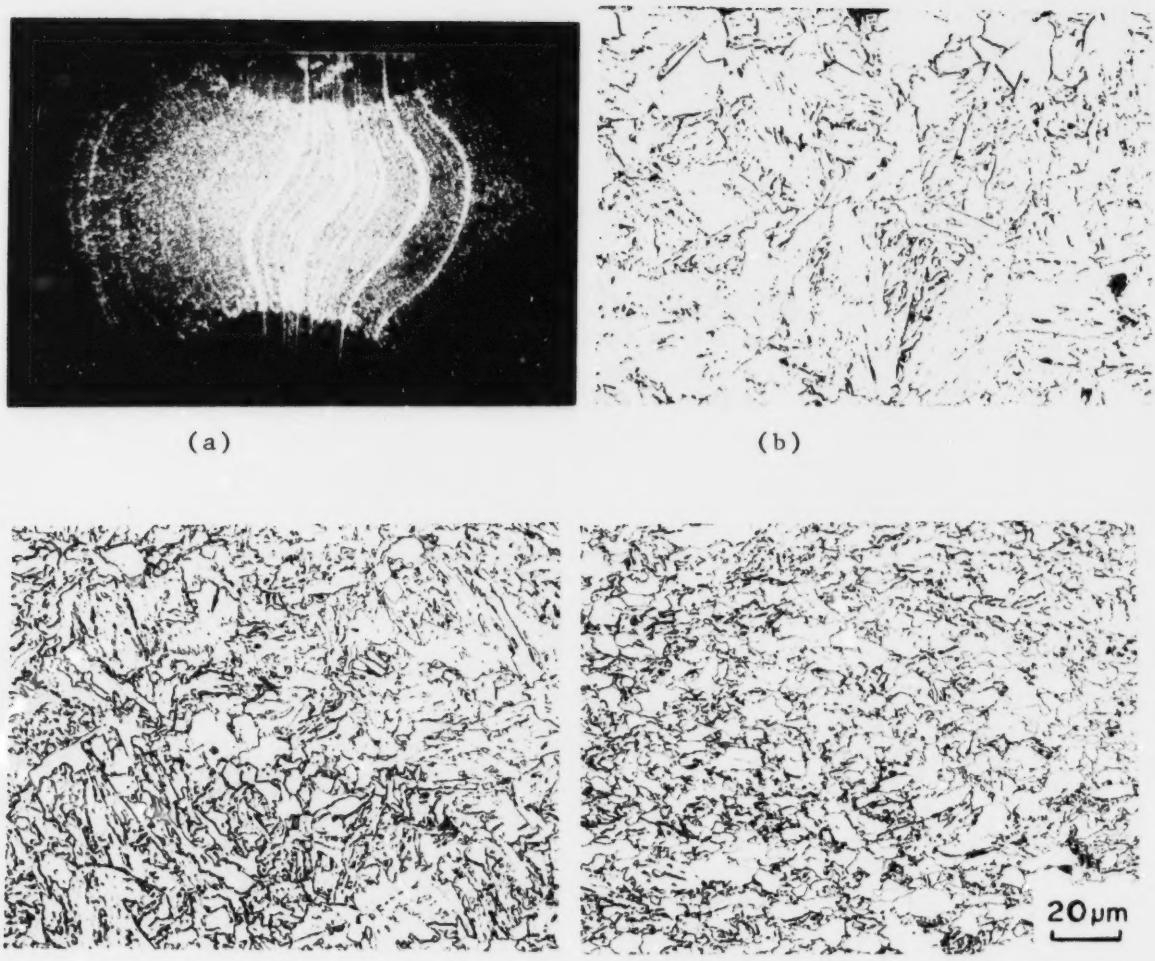


Fig. 7. Effect of gas flow rate on cooling time between 800 and 500°C for the a) standard specimen size (6 mm diam) (b) specimens deformed 50% (4 mm initial diam). Open symbols indicate Ar cooling and closed symbols indicate He cooling. (neg 10665, 10666)



(a) (b)

(c) (c)

Fig. 8. (a) Barrelled sample showing inhomogeneous deformation pattern.
(b) Microstructure of steel B deformed by 50% and cooled such that $T_{800\text{-}500°C}$ = 100 s - top surface.
(c) Same as (b) - ¼ h.
(d) Same as (b) - center line.

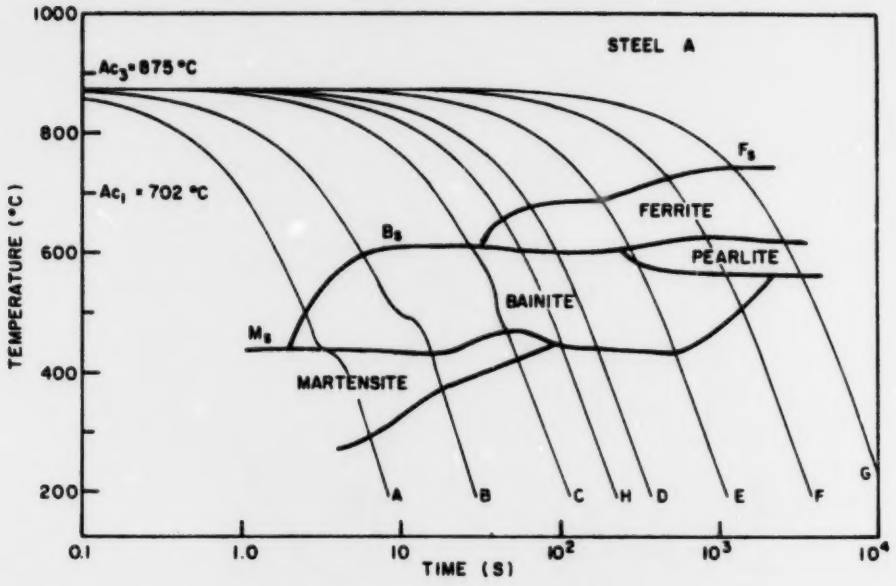


Fig. 9. Cooling rates used for determination of a CCT diagram. (neg 10668)

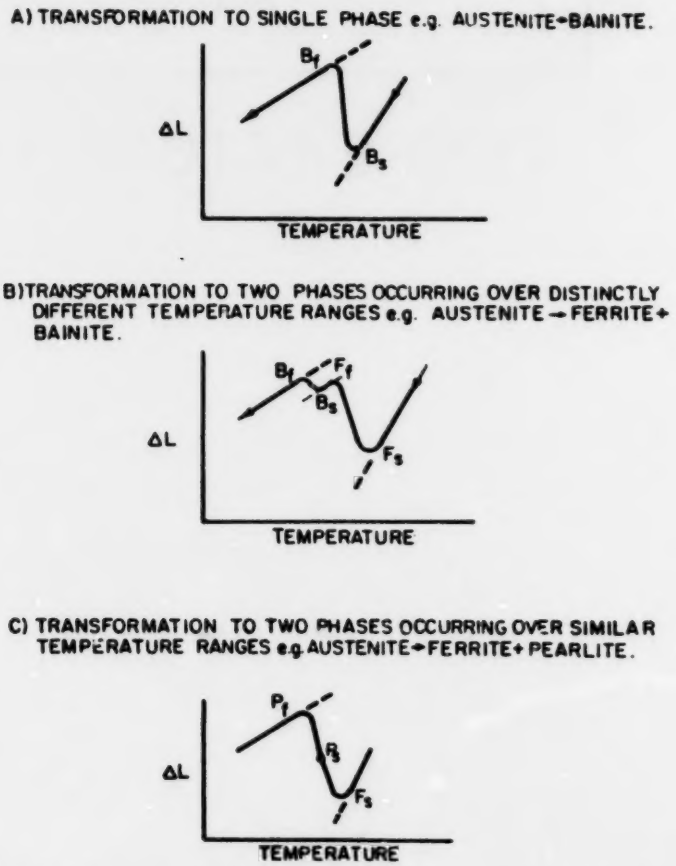


Fig. 10. Determination of transformation start and finish temperatures. (neg 10671)

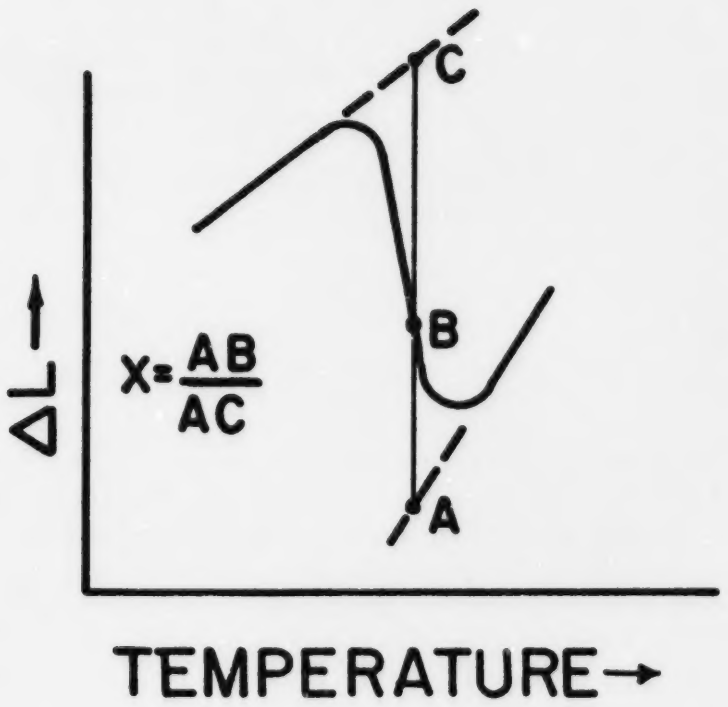


Fig. 11. Determination of fraction transformed. (neg 10673)

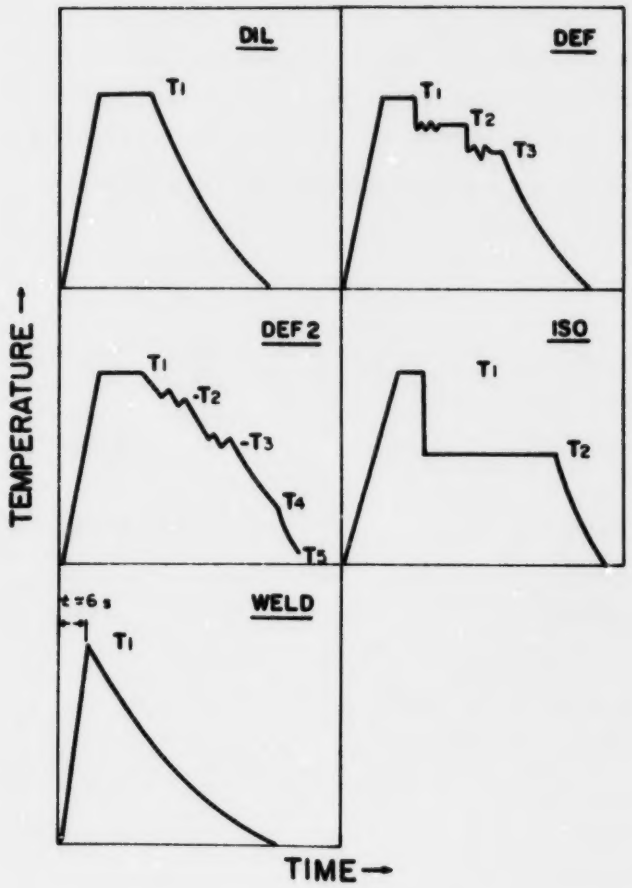


Fig. 12. Illustration of thermomechanical treatments available with computer control. (neg 10669)

Appendix I

Determination of Ac_1 and Ac_3

The program for the dilatometer microprocessor used to determine the Ac_1 and Ac_3 temperatures is listed below. The specimen is heated to 500°C in 1 min and then at 2°C/min to 1000°C to allow determination of Ac_1 and Ac_3. On reaching 1000°C the specimen is quenched to room temperature.

STEP	ENTRY	OPERATION
	EN'BL ENTRY	
	ENT MAN	
	1999 ENT PROG	-clears memory
1	1 ENT PROG	-initiates program
	1 ENT SEC	
	NEXT POINT	
2	5 ENT SEC	-pause
	NEXT POINT	
3	25.2 ENT SP1	-heat to 500°C in 1 min
	1 ENT MIN	
	6 ENT E-ON	-pen down
	NEXT POINT	
4	50.4 ENT SP1	-heat to 900°C at 2°C/min
	200 ENT MIN	
	NEXT POINT	
5	57.2 ENT SP1	-heat to 1000°C at 2°C/min
	50 ENT MIN	
	NEXT POINT	

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6       1 ENT MIN
        6 ENT E-OFF                  -lift pen on X-Y recorder
        NEXT POINT

7       30 ENT SEC
        1 ENT E-ON                   -close high vacuum valve
        NEXT POINT

8       0  ENT SP1                  -cool to R.T.
        30 ENT SEC
         1 ENT E-OFF
         2 ENT E-ON                  -CCT cooling
         7 ENT E-ON                  -furnace off
         6 ENT E-ON                  -pen down on X-Y recorder
        NEXT POINT

9       2 ENT E-OFF                  -shut off gas
        5 ENT E-ON                   -stop chart and lift pen on
                                      strip recorder
        6 ENT E-OFF                  -lift pen on X-Y recorder
        2 ENT SEC
        NEXT POINT

10      10 ENT NEXT POINT            -stop program
        NEXT POINT

        1 ENT RUN                    -starts program
```